DUBBLE experiment 26-01-976 experiment report

Building of the confocal setup for fluorescence mode µ-XANES, consisting of a focussing polycapillary optic in the incident X-ray beam path and a collimating polycapillary optic in front of a Vortex-EM SDD detector, was challenging because experienced experimentalists who were assigned previously to the project of upgrading the DUBBLE spectrometer for microscopic 3D-XANES are not available anymore. Additionally, several motors used in the setup had insufficient precision or were malfunctioning, preventing an optimal alignment of the highly sensitive confocal setup.

The investigated samples were iron containing (~1 mol%) alkali-aluminosilicate glasses, before and after Na<->K ion exchange (IOX). The goal was to study the variation of iron oxidation and coordination states as a function of depth inside the glass. The confocal volume was characterized using several standard reference materials including thin metal wires (e.g. Goodfellow 10 µm stainless steel wire) and metal foils. Given the difficulties of the experimental setup described above, an optimal confocal volume of $20(H)\times20(V)\times40(D)\ \mu m^3$ was obtained.

A sample depth scan with 10µm step size was performed through the confocal volume, performing fluorescence XANES measurements at each step, extracting information from depths up to 90 µm. Pre-edge structure study and linear combination fitting of the spectra using reference iron oxide compound spectra, lead to the conclusion that iron is mainly present as Fe^{3+} in both IOX'ed and non-IOX'ed samples. The non-IOX'ed glass does not show a fluctuation in iron oxidation or coordination state as a function of depth, as was expected. In the IOX'ed glass, a variation may be observed in iron coordination state towards lower coordination number. In-depth pre-edge structure study is however complicated due to poor counting statistics and too low energy resolution in this energy region. Further experiments focussing on this energy region with better counting statistics and better energy resolution are necessary to confirm these results. Additionally, the detailed documenting of the experimental difficulties that should be improved will result in faster and more efficient setup building and alignment in the future.